

# Monday Morning, November 10, 2014

## Vacuum Technology

Room: 303 - Session VT-MoM

## Vacuum Measurement, Calibration, and Primary Standards

**Moderator:** Steve Borichevsky, Applied Materials, Varian Semiconductor Equipment, Yulin Li, Cornell University

8:20am **VT-MoM1 Miniature Fiber Optic Pressure Sensors: Technologies and Applications**, *Miao Yu*, University of Maryland, College Park **INVITED**

Compared with their electrical counterparts, fiber optical sensors offer many advantages including small size, immunity to electromagnetic interference, convenient light guiding through optical fibers, high sensitivity, high resolution, large bandwidth, and low noise. Recently, miniature fiber optic pressure sensors have attracted much attention for many applications including biomedical, surveillance, and industrial applications. Most of these sensors are based on a Fabry-Perot interferometer fabricated directly on an optical fiber end face with a silica/silicon (Si) diaphragm serving as a pressure transducer. Although these sensors have a small size (~100s microns in diameter), they suffer from important issues including low sensitivity due to the high elastic moduli of silica/Si (130-185 GPa), and high brittleness and easy breakage of the sensor elements.

In this paper, the research program at the Sensors and Actuators Laboratory (SAL) of the University of Maryland on miniature fiber optic pressure sensors based on polymer or graphene diaphragms will be introduced. First, our work on polymer based miniature optical pressure sensors will be discussed. The polymer materials have superior elasticity and high fracture strength, which enables polymer based sensors to have superior sensitivity even at a small size and helps prevent cracking or breaking of the sensors. Furthermore, these sensors can be fabricated by using relatively simple and inexpensive processes. We have developed several unique low-cost micro-fabrication processes for these sensors, including self-aligned photolithography and UV molding process. By using simple and safe procedures, a polymer based Fabry-Perot cavity can be directly fabricated at the end of optical fiber, thus eliminating the necessity for complicated assembly of the sensing element and the optical fiber. Further, since polymer based sensors inherently suffer from temperature drift due to the large thermal expansion coefficient of polymer materials, novel temperature compensation methods for the polymer based fiber optic pressure sensors will also be discussed. Second, miniature fiber optic pressure sensors utilizing a graphene diaphragm will be presented. Graphene is believed to be one of the strongest materials and the thinnest film in the universe, and it can be stretched by as much as 20%. These unique mechanical properties render graphene an excellent choice for miniature acoustic sensors with unprecedentedly high sensitivity, large bandwidth, and large dynamic range. Finally, the potential applications of these different sensors will be discussed.

9:00am **VT-MoM3 Quantum Based Vacuum Standard**, *Jay Hendricks, J.A. Stone, J.E. Ricker, P.F. Egan, G.E. Scace, D.A. Olson*, National Institute of Standards and Technology, *D.R. Gerty*, Sandia National Laboratories, *G.F. Strouse*, National Institute of Standards and Technology  
The future of pressure and vacuum measurement will employ lasers, Fabry-Perot optical cavities, and quantum physics. Photons interact at the quantum level with matter such that light travels at a slower speed in gas than it does in vacuum. NIST is developing a fixed length optical cavity (FLOC) and variable length optical cavity (VLOC) that will make simultaneous ultra-precise measurements of vacuum and gas cavity photon-path-lengths. While pressure is a widely measured unit in every day processes, the standard on which it is based, the mercury manometer is quite old and traces its early beginnings to 1643. In the future, the mercury barometer will be replaced with a new standard based on quantum chemistry calculations of helium's refractive index. This will enable the replacement of all artifact-based mercury standards. Measuring pressure optically represents a paradigm shift in the way the unit is realized and will move us from a primary standard based on an artifact to a primary standard based on quantum-chemistry calculations of helium's refractive index. This talk will cover current status and early prototype results of NIST's Innovations Measurement Science (IMS) project (the second year of five) that will have profound impacts on how pressure, temperature and length in air measurements are made in the future. While the primary aim of the project is to create new measurement infrastructure for NIST, it will also create exciting spin-off technology that will have large impacts for US manufacturing and world metrology.

9:20am **VT-MoM4 New PTB Standard to Provide Traceability for Partial Pressure Measurement**, *Karl Jousten*, Physikalisch-Technische Bundesanstalt (PTB), Germany

Partial pressure measurement in vacuum by quadrupole mass spectrometer (QMS) is an important tool to control and monitor processes in industry and to measure outgassing rates. It is, however, difficult to obtain reliable results with quadrupole mass spectrometers, since its calibration is ill-defined and at present there is no traceability to any national primary standard.

PTB has recently established a new calibration system to calibrate QMS. It is based on the continuous expansion method and allows generating three partial pressures at the same time in the calibration chamber with pressure ratios between them of a factor of up to  $10^8$  with the uncertainty of each partial pressure depending on the value and the gas species. In the minimum the uncertainty of partial pressure is close to 1%. With less accuracy more gas species than three can be mixed.

The flow of desired gas into the calibration chamber is generated by nano-holes, glass capillaries or sintered elements characterized in terms of conductances for some gas species. Since the flow through these conductance elements is of molecular type up to about 10 kPa, the flow can be predicted for any gas species.

Four QMS were characterized with the new system and some results will be given. The first goal with this standard is to test and establish calibration procedures for written standards for several parameters of QMS like sensitivity, minimum partial pressure and minimum concentration.

Support through the EMRP IND12 project is gratefully acknowledged. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

9:40am **VT-MoM5 Study of Long Term Stability of Quadrupole Mass Spectrometers**, *Janez Setina*, Institute of Metals and Technology (IMT), Slovenia, *A. Elkatmis, R. Kangi*, Ulusal Metroloji Enstitüsü (TUBITAK UME), Turkey, *K. Jousten*, Physikalisch-Technische Bundesanstalt (PTB), Germany, *M. Bergoglio*, Istituto Nazionale di Ricerca Metrologica (INRIM), Italy, *F. Boineau*, Laboratoire National de métrologie et d'Essais (LNE), France, *S. Ruiz*, Centro Español de Metrología (CEM), Spain, *M. Vicar*, Czech Metrology Institute (CMI), Czech Republic

Quadrupole mass spectrometers (QMS) are widely used in industry for leak detection, residual gas analysis and measurements and control of gas composition in vacuum processes, which require well defined partial pressures of different gases in low pressure environment. Consistent measurements and process stability are possible solely if measurement instrumentation is sufficiently stable with time. Only few studies about the time stability of QMS have been reported in the literature. They mainly indicate that QMS instruments are less stable than typical Bayard-Alpert ionization gauges, so frequent recalibrations may be required.

To get information about typical quality of commercial instruments, a group of European national metrology institutes performed a joint study of time stability of some metrological characteristics of seven different QMS. Parameters under study included: sensitivity for gases He and N<sub>2</sub>, mass resolution, mass scale stability, secondary electron multiplier (SEM) gain, and minimum detectable partial pressure. Typical check intervals were 3 months. Study started in the middle of 2012 and the overall duration was two years.

In 3 months periods the typical changes of sensitivity between 10% and 30% were observed. The peak positions were stable within 0.05 amu and 0.2 amu and mass resolution was stable within 0.02 and 0.05 amu. For most of instruments a gradual decrease of the SEM gain as a result of aging of the multiplier was observed. For some instruments the SEM gain dropped by more than 40 % in 2 years period.

Support through the EMRP IND12 project is gratefully acknowledged. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

10:00am **VT-MoM6 The Stability of Spinning Rotor Gauges as Transfer Standards**, *James Fedchak*, National Institute of Standards and Technology (NIST)

The spinning rotor gauge (SRG) has long been used as a transfer standard for high vacuum calibrations and in national and international intercomparisons of high-vacuum standards. It is typically used over a pressure range of  $1 \times 10^{-4}$  Pa to 1.0 Pa ( $10^{-6}$  Torr to 10 mTorr) and tends to be favored by metrology and calibration laboratories because of its reputation as being a very stable standard. Here we will review the stability data for SRGs used in international key comparisons. Different rotor

materials and various transfer techniques have been employed in these comparisons. These will be discussed in context of the best practices and techniques that have produced the best rotor stability. Various factors which may affect rotor stability will also be discussed, and data taken at NIST as well as that found in the literature will be presented. The goal of this study is to establish the best practices for the use of a spinning rotor gauge as a transfer standard, as well as establishing the best stability that can be reasonable achieved.

10:40am **VT-MoM8 Pilot Study for International Comparison of Absolute Pressure Measurement from  $3 \times 10^{-9}$  Pa to  $9 \times 10^{-4}$  Pa, Hajime Yoshida, K. Arai, E. Komatsu, K. Fujii**, National Institute of Advanced Industrial Science and Technology (AIST), Japan, *K. Jousten, T. Bock*, Physikalisch-Technische Bundesanstalt (PTB), Germany

International comparison of absolute pressure measurements in gas from  $3 \times 10^{-6}$  Pa to  $9 \times 10^{-4}$  Pa, identified as CCM.P-K3, was performed from 1998 to 2002 to determine the degree of equivalence of national metrology institutes (NMIs). A new international comparison, where the pressure range is expanded down to  $3 \times 10^{-9}$  Pa, is planned as CCM.P-K3 follower. A pilot study was performed in advance because two challenging issues are including the CCM.P-K3 follower. One is the stability of extreme high vacuum (XHV) gauge as a transfer standard. XHV gauges are kinds of hot cathode ionization gauges with a structure to reduce disturbances such as X-ray, electron stimulated desorption (ESD) ion, and so on. Few experiments are reported about the stability of XHV gauges from the viewpoint of metrology. The other is the stability of transfer gauges against to repeating bake-out because baking the calibration chamber including transfer gauges is inevitable to achieve XHV.

At first, three types of XHV gauges; Axial-symmetric transmission gauge (ATG), extractor gauge (EXG), and bent belt-beam gauge (3BG) were tested in NMIJ. Stabilities against air exposure and following bake-out were also tested for both XHV gauges and spinning rotor gauges (SRGs). A protocol for pilot study from  $3 \times 10^{-9}$  Pa to  $9 \times 10^{-4}$  Pa was prepared based on the results of both these tests and previous CCM.P-K3. ATG and EXG were adopted as transfer gauges because of achievements so far, although the stabilities of tested XHV gauges were comparable. In addition, two SRGs (SRG-1 and SRG-2) were also adopted as transfer gauges to calibrate at  $9 \times 10^{-4}$  Pa because stabilities of SRGs were expected to be better than those of XHV gauges. Measurement results of XHV gauges are normalized by those of SRGs. No significant differences of stabilities were observed whether the calibration gas was  $N_2$  or Ar.  $N_2$  was selected as the calibration gas because  $N_2$  is typical calibration gas for ionization gauges.

A bilateral comparison between NMIJ and PTB was performed from May 2013 to Jan 2014 to confirm the effectiveness and to test the transport stability of the transfer standards. Shift of sensitivities of ATG, EXG, SRG-1, and SRG-2 were less than 0.58 %, 0.67 %, 3.5 %, and 0.28 %, respectively. The results of comparison were summarized except for data of SRG-1 because it clearly shows the drift of the sensitivity (effective accommodation coefficient). Results of the comparison show good agreement within the claimed uncertainty. Details will be presented at the conference.

11:00am **VT-MoM9 Stability of the Cold Cathode Ionization Gauge, Paul Arnold, G.A. Brucker**, Granville-Phillips Vacuum Products

A study of the stability of cold cathode ionization gauges (CCIG) using an evaluation of physics principles affecting the ionization properties of the CCIG has been performed. The variation of the response to pressure of the CCIG is significantly dependent upon the interaction of the plasma discharge with the interior surfaces of the CCIG occurring during operation of the CCIG. New investigations will demonstrate these effects. The concept of measured pressure dose provides an index of likely drift in CCIG performance resultant from the magnitude of the plasma interaction, including the concept of remaining gauge useful life. The nature of the above plasma interaction changes the magnetic field internal to the CCIG as well as electron production from the cathode electrode, resulting in drift in CCIG performance. Both simulation and test data of these phenomena will be presented, showing relation of pressure dose to pressure performance with explanation of mechanisms.

11:20am **VT-MoM10 Cold Cathode Ionization Gauge Design Mitigates Well-known Performance Issues, Brandon Kelly, G.A. Brucker**, Granville-Phillips Vacuum Products

Cold Cathode Ionization Gauges (CCIGs) are a well established indirect pressure measurement tool that has well documented advantages and shortcomings. In an attempt to overcome these design challenges a series of experiments have been conducted exploring different electrode geometries and configurations. Included in these experiments are high power magnet assemblies with unique discharge chamber geometries aimed at increasing device sensitivity at UHV pressures. Various modifications and methods

have been tested to control the plasma discharge interaction with the interior of the CCIG for which increased lifetime will be shown. In addition to gauge sensitivity, other common shortcomings are addressed such as high vacuum starting time statistics and the separation of leakage current from plasma (i.e. discharge) current. A novel ionization chamber design prevents the escape of material from the internal surfaces of the gauge while preserving adequate conductance to the vacuum chamber and providing the ability to replace parts in the field. The theory and details of these new sensor design modifications will be discussed offering an insight into the next generation of cold cathode ionization gauges.

11:40am **VT-MoM11 A Systematic Study of Long-Term Vacuum Gauge Performance, Gerardo Brucker, S. Heinbuch, T.C. Swinney**, Granville-Phillips Vacuum Products

Cold Cathode Ionization Gauges (CCIGs) have been the subject of numerous investigations throughout several decades. The free-running characteristics of their pure electron plasma, combined with the in-depth investigation of this phenomenon interacting with the interior of these devices, have led to numerous theories and speculation regarding the factors that regulate the long term performance of the gauges. Our laboratory has become increasingly interested in extending gauge lifetime through improved design and in the development of innovative dose calculation methodologies that can provide an adequate estimation of gauge remaining lifetime. We are particularly interested in understanding the phenomena that lead to drifts in gauge sensitivity with dose and specific gas chemistry. During our long term studies, gauge sensitivity is tracked over time against dose and the most modern surface and chemical analysis methodologies are employed to detect and understand the physico-chemical changes that take place at the internal electrodes. In this presentation we will demonstrate that while attempting to understand changes in gauge sensitivity it is equally important to consider changes in the magnetic as well as the internal electric characteristics of cold cathode gauges. Prior work by other groups has focused heavily on the influence of surface chemistry modifications. Our recent work will demonstrate that equal attention must be paid to the internal magnetic properties to fully account for long term changes in sensitivity.

# Monday Afternoon, November 10, 2014

## Vacuum Technology

Room: 303 - Session VT-MoA

## Vacuum Measurement, Applications of UHV and Ultraclean Processes

**Moderator:** Joe Becker, Kurt J. Lesker Company, Bob Garcia, SAES Getters

2:00pm **VT-MoA1 A Capacitance Diaphragm Gauge with 10 mTorr Full Scale**, *Martin Wüest*, INFICON Ltd., Liechtenstein, *P. Björkman, J. Bäckman*, INFICON Ab, Finland

Etching of semiconductors chips is a complex process and there are critical process steps that tend to go to lower pressures. At lower pressures scattering at the residual gas is reduced which allows obtaining narrower and deeper vias, important for three-dimensional chip structures. Many etch process pressures today are around 1 Pa and use a 13.3 Pa full scale CDG in the second measurement decade where accuracy is already lower. We use ceramic technology to build pressure sensors that measure an elastic deformation of a diaphragm under the feeble pressure forces while being highly resistant to etch chemistries. Improvements to the manufacturing process now allow us to manufacture thinner, highly elastic, leak tight membranes. We now have developed a heated capacitance diaphragm gauge with 1.33 Pa (10 mTorr) full scale. The low full scale is achieved by the deflection of a thinner membrane and focus on noise reduction and not by electronic amplification. We will present pertinent performance parameters.

2:20pm **VT-MoA2 What if Saving Energy become Important on Bayard Alpert Hot Ionization Gauges?**, *Simon Naef*, INFICON Ltd., Liechtenstein

Hot ionization gauge have been on the market for decades and many refinements have been made over the time. A lot of common knowledge how to build a stable, sensitive, and accurate BA-gauge is recorded. How about overall power consumption and the energy efficiency of hot ion gauges? The topic of energy conservation has been neglected so far.

The saved energy per gauge is not essential, but on the global view the power consumption can be reduced significantly. Even the high energy consuming semiconductor industry tries to reduce their footprint, since 80% of energy used in this industry is manufacturing and transportation.

How can that be achieved? Integration and miniaturization is way used in the past. So there are many compact hot ion gauge designs available on the market today, which use obviously less energy than larger full-size hot ionization systems. But what is drawback of going small and is worth going small?

On one hand the heated cathode defines mainly the power consumption, which is based on the electron emissivity of the surface material used and the resistance of the used wire material. On the other hand the sensitivity of BA-gauge is based on the geometry of the electrodes, which can be correlated to the efficiency of the gauge. Is there any option with any optimal compromise size?

3:40pm **VT-MoA6 How to Create as Less as Possible to Make the Best as Possible**, *Norbert Koster*, TNO Technical Sciences, Netherlands  
**INVITED**

The field of vacuum technology is rapidly becoming a full blown industry, with parts and component suppliers, OEM manufacturers and end users (IDM). The generation of vacuum is not the goal, but the means to realize products and devices. Especially the semiconductor industry is one of the driving forces in this development, but also large scientific projects like ITER, Cern and others require a supply chain that can deliver parts that meet the requirements and certification. The upcoming introduction of EUV lithography required a change in the supply chain for manufacturers like ASML with suppliers that had to deliver vacuum qualified assemblies without any knowledge of vacuum technology.

The introduction of EUV also meant very complex vacuum systems that could not be baked anymore but with cleanliness demands that superseded the cleanliness that can be achieved with baked UHV systems. This was achieved by a rigorous cleaning and qualification process, standardized outgassing measurements and budgeting of all the parts and assemblies. To make a distinction between ordinary type of vacuum system building and this new way of making vacuum systems the phrase Ultra Clean Vacuum (UCV) was introduced. This presentation will describe how we solved a number of problems that occurred during this type of manufacturing, this includes supply chain engineering, cleaning procedures and solutions in the

tool itself. This way of creating vacuum systems is now also being used by other OEM's like AMAT, KLA-Tencor who are building highly complicated metrology tools and processing equipment, while vacuum generation is not their core business.

4:20pm **VT-MoA8 Study of Potential Particle Generation by Ion Sources During EUV Mask Blank Deposition**, *Ivan Shchelkanov, A.M. Lietz, J. Pachicano*, University of Illinois at Urbana-Champaign, *A. Antohe, P. Kearney, SEMATECH, D.N. Ruzic*, University of Illinois at Urbana-Champaign

We summarise the research accomplishments of the CPMI-SEMATECH research project "Identification of possible defect sources and particle characterisation from sources used in EUVL chambers for mask blank manufacturing". The main goal of the project is the determination of nanoparticle production (100-350 nm in size) from the ion source currently used for mask blank manufacturing in EUV lithography chambers at SEMATECH. Combination of Volumetric Laser Scattering and Surface Laser Scattering system, and Scanning Electron Microscopy (SEM) was used to develop time resolved phenomenological model of nano-particle generation inside vacuum chamber during ion beam source operation. In this project a Veeco 3cm RF ion beam source with two molybdenum grids and RF neutralizer was investigated. Stiletto Volumetric Laser Scattering system developed by INFICON together with Surface Laser Scattering system, developed at CPMI were used to detect nano-particles. This combination of laser systems was able to detect particles with the size of 100 nm and bigger. Scanning Electron Microscopy was used to track evolution of the ion source grids surface. SEM photos of the grids and polished silicon wafers on the ion beam path were used to identify and characterize nano-particles via size, shape and material. Nano-particle generation rate by the ion beam source was measured and recommendations for the source operation are made.

4:40pm **VT-MoA9 Particle Defect Reduction in EUV Mask Blank Production Devices**, *Amanda Lietz, I.A. Shchelkanov*, University of Illinois at Urbana-Champaign, *A. Hayes, Veeco Instruments, Inc., J. Pachicano, S. Keniley, D.N. Ruzic*, University of Illinois at Urbana-Champaign

Extreme UltraViolet Lithography (EUVL) requires reflective mask blanks, manufactured by ion beam sputtering a multilayer stack of thin films, primarily Mo and Si, onto a mask substrate. At least 40 bilayers of Mo and Si are necessary to produce a surface which has sufficient EUV light reflectivity for use in high volume manufacturing exposure tools. When contaminant particles deposit between these layers, the EUV light is absorbed or scatters irregularly, rendering the mask blank unusable. One possible source of such particles is bombardment of shields in the deposition chamber by energetic particles scattered from the ion beam and target and "overspill" of the tails of the ion beam off the edge of the target. Stainless steel shields are used to cover targets that are not in use and prevent deposition or sputtering nearby surfaces and equipment. These shields must be able to accept many successive layers of deposition without flaking and forming particles of deposited material. They also must be able to withstand ion beam overspill bombardment, while forming a minimal amount of particles.

In order to evaluate improved shield materials and surface finishes, shield samples of various treatments were placed under a broad angle ion beam and particles were collected on a witness plate. The total number of particles on the witness plates was quantified using laser scattering particle detection which was capable of detecting particles greater than 125nm in size. Etching treatments of the shields show an improvement from 13.0±.7 particles/mm<sup>2</sup> (for untreated steel) to .022±.016 particles/mm<sup>2</sup> (for etched steel). Shields of various materials and surface finishes were compared to determine the lowest level of particle formation

5:00pm **VT-MoA10 VTD Early Career Award: Novel Vacuum Processing of Thin-Film Photovoltaic Materials**, *Jason D. Myers\**, *J.A. Frantz, R.Y. Bekele, V.Q. Nguyen, C.C. Baker, S.C. Erwin, N.D. Bassim*, U.S. Naval Research Laboratory, *A. Bruce, S.V. Frolov*, Sunlight Photonics, *J.S. Sanghera*, U.S. Naval Research Laboratory  
**INVITED**

In this presentation, two different avenues of research into thin film photovoltaics will be discussed. The first part of the talk will be focused on quaternary-sputtered Cu(In,Ga)Se<sub>2</sub> (CIGS) thin film photovoltaic devices. Current state-of-the-art CIGS devices are produced using a multistage thermal coevaporation process that has resulted in laboratory efficiencies in excess of 20%, but this process is difficult to implement at a commercial

\* **VTD Early Career Award**

scale. Our work has instead focused on developing a scalable deposition technique using RF magnetron sputtering of quaternary CIGS. The resulting films do not require post-selenization, reducing processing time and cost. We have fabricated devices above 10% efficiency using this approach, showing its promise as a production method for high-performance CIGS.

The second part of the talk will be focused on an emerging thin film photovoltaic system, FeS<sub>2</sub>. Based on its favorable bandgap, high absorption coefficient, and immense earth abundance, FeS<sub>2</sub> is a highly promising material for grid-scale energy production. However, no successful thin-film photovoltaic devices have been realized due to surface defect states that arise due to the cubic pyrite structure, where sulfur atoms are differentially bonded at the surface compared to the bulk; this leads to extremely low open circuit voltages and poor diode characteristics. To solve this issue, we are developing vacuum-deposited inorganic capping films to heal these defects by providing bulk-like coordination at the FeS<sub>2</sub> surface. FeS<sub>2</sub> films with ZnS capping layers show a significant decrease in surface state character, an important step towards efficient FeS<sub>2</sub> photovoltaics.

# Tuesday Morning, November 11, 2014

## Applied Surface Science

Room: 316 - Session AS+BI+VT-TuM

### Ambient Ionization Mass Spectrometry

**Moderator:** Gerardo Brucker, Granville-Phillips Vacuum Products, Steven Pachuta, 3M Company

8:00am **AS+BI+VT-TuM1 Laser Ablation Electrospray Ionization Mass Spectrometry with Ion Mobility Separation for Cell and Tissue Analysis**, *Akos Vertes, B. Shrestha, H. Li, S.A. Stopka, L. Zhang*, George Washington University **INVITED**

Laser ablation electrospray ionization (LAESI) is a novel ion source that enables the direct analysis of biological samples, including tissues and individual cells. In this ionization method, mid-IR laser ablation is followed by electrospray ionization of the ablated material in the expanding plume. Molecular coverage in complex biological samples is limited, in part, by the large number of components and the absence of a separation step prior to ionization. In addition, isobars, such as structural isomers and conformers, are not distinguished by mass analysis alone. To overcome these limitations, LAESI is combined with ion mobility separation (IMS) before mass spectrometry (MS). In this contribution, we describe the first results with such a LAESI-IMS-MS system for metabolite, lipid and protein analysis, including its application to plant and animal tissues, MS imaging and single cell analysis. The studied systems, among others, comprise mouse brain sections, *Arabidopsis thaliana* leaves and green algae (*Chlamydomonas reinhardtii*) cell pellets. The introduction of IMS resulted in enhanced molecular coverage, reduced interferences, distinction of structural isomers, observation of larger multiply charged ions typically suppressed by singly charged abundant metabolites and phospholipids, and in extended dynamic range.

8:40am **AS+BI+VT-TuM3 Miniature Mass Spectrometry Systems with Ambient Ionization and MS/MS Capabilities**, *Zheng Ouyang, L. Li, Y. Ren, X. Wang, X. Ma, R. Zou, R.G. Cooks, Y. Xia*, Purdue University **INVITED**

As a technique for chemical analysis, mass spectrometry is versatile and provides very specific information. High sensitivity can be achieved when sample matrix effect is properly suppressed. Miniaturization of the mass spectrometry instrument system and simplification of the operation procedure enable the chemical analysis outside the analytical laboratories and/or by personnel without special trainings. The development of these systems goes beyond the miniaturization of the mass analyzers and mass spectrometers. At Purdue, we have taken an approach of combining the ambient ionization for direct sampling and the miniature ion trap mass spectrometer with MS/MS capability. The miniature systems use linear ion traps (LIT) for mass analysis and can perform multi-stage MS/MS, which help to improve the specificity of the analysis using the fragmentation pattern of the target analyte and to eliminate the chemical noise from the complex mixtures. A discontinuous atmospheric pressure interface (DAPI) has been developed to allow coupling of ionization sources at atmospheric pressure with the instruments using miniature pumping systems to support the vacuum. The DAPI opens for about 20 ms for ion introduction and requires a 200 ms delay for pressure drop prior to mass analysis. The complex gas dynamics has been characterized using direct simulation Monte Carlo method and an electro-hydrodynamic simulation method has been developed for predicting the ion trajectory for DAPI instrument design. While mass spectrometers as light as 4 kg have been previously developed with capability of analyzing non-volatile compounds, two complete MS analytical systems have recently developed as the backpack MS for in-field analysis and the Mini 12 desktop system for point-of-care analysis by nurses and physicians. These two systems use ambient ionization for direct sampling analysis. The low temperature plasma (LTP) probe was modified with an in-line configuration for point-and-shoot operation with the backpack MS. New ambient ionization methods have been explored for development consumable sample cartridges for the Mini 12 system, which include the paper spray, extraction spray and the most recent slug flow microextraction nanoESI. IS-coated capillary samplers have been developed for highly quantitative analysis using several microliters of biofluid samples and extremely operation procedures. On-cartridge chemical derivatization has been developed to significantly improve the sensitivity of the target analytes in complex biological samples and on-cartridge assays have also been studied for direct monitoring the enzymatic functions. Direct analysis of the biological tissues have also been explored using Mini 12 and on-line Patenó-Büchi (P-B) reactions facilitated by UV irradiation has also been implemented to identify the locations of

C=C bonds in the lipids, which is highly relevant to the biosynthetic pathways and the function of the lipids. The relative ratios of the unsaturated isomers can now be quantified, as the potential biomarkers for diagnosis of diseased tissues.

9:20am **AS+BI+VT-TuM5 The Importance of Sample Form and Surface Temperature for Analysis by Ambient Plasma Mass Spectrometry (PADI)**, *Ian Gilmore, T.L. Salter, J. Bunch*, National Physical Laboratory, UK

Plasma sources for ambient mass spectrometry are of increasing importance owing to their ability to analyse a wide range of organics including polymers. Some industrially important molecules are not successfully analysed by electrospray based methods and here plasma methods are making an important contribution. For analysis in industry, it is essential to understand the fundamental mechanisms so that predictions can be made of which types of materials can and cannot be detected. In this study, we develop a metrology framework to understand the sensitivity of PADI to different substances and material form. We study in detail, the effect of sample temperature on the signal intensity and show that the intensity is proportional to the vapour pressure. Importantly, we also show the sample form, as a film or powder, has a strong effect of sensitivity. For the analysis of thin films at room temperature and using a low plasma power, a vapour pressure of greater than  $10^{-4}$  Pa is required to achieve a sufficiently good quality spectrum. Using thermal desorption we are able to increase the signal intensity of materials with vapour pressures less than  $10^{-4}$  Pa, in thin film form, by between 4 and 7 orders of magnitude. This is achieved by increasing the temperature of the sample up to a maximum of 200 °C. Thermal desorption can also increase the signal intensity for the analysis of powders. Prospects for imaging PADI and sub-micron imaging ambient mass spectrometry imaging will also be discussed.

9:40am **AS+BI+VT-TuM6 A VAMAS Interlaboratory Study for Desorption Electrospray Ionisation Mass Spectrometry (DESI MS) - Survey of the Measurement Issues**, *Paulina Rakowska, E. Gurdak, F.M. Green, M.P. Seah, T.L. Salter, I.S. Gilmore*, National Physical Laboratory, UK

The DESI technique is celebrating a decade of application since its innovation in 2004. There has been significant progress in understanding its fundamentals and a rapid expansion in the applications, covering a diverse range of science and technologies. For wider uptake in industry, measurements need to be repeatable and constant. It is especially important to test that methods are transferable between different instrument designs and that analytical procedures are clear. This requires the development of a metrological infrastructure. Interlaboratory studies are an effective route to do this. VAMAS provides an excellent mechanism for such evaluation. Under this framework, the National Physical Laboratory (UK) has conducted a DESI interlaboratory comparison. The objectives of this study were to determine the current achievable repeatability and constancy of instruments. The comparison was conducted with the involvement of 20 laboratories from 10 different countries. The instruments used included 7 commercially made DESI sources with the remainder home-built. A variety of mass spectrometers were used including 13 Ion Traps, 4 Orbitraps and 4 Time-of-Flight. Participants were provided with an analytical protocol and two reference samples: a thin layer of Rhodamine B and a double-sided adhesive tape. The studies comprised acquisition of positive ion mass spectra in pre-determined  $m/z$  ranges. No sample preparation was required. Results for Rhodamine B show that intensity repeatabilities below 20 % may be achieved. However, inadequacies of the spray and sample stage designs lead to repeatabilities that average 50 % with some worse than 80 %. Rhodamine B is an excellent reference sample to check the sample erosion, the sample stage movement and memory effects. The adhesive tape samples show that the absolute intensity repeatability is 31 % with several achieving below 20%. Importantly, the spectral response, given by the relative repeatability, not measurable with Rhodamine B, was reduced to 9 % with a significant number achieving the 5 % expected of more mature analytical methods. The constancy of these spectra from relative intensities gives day-to-day averages of 31 %, over three times worse than the short term repeatability. Significant differences in the spectra from different laboratories arise from different factors. This first interlaboratory study has provided an effective survey of the measurement issues and some important conclusions can be drawn about the possibilities for DESI MS concerning overall practice, reference samples and recommendations for the future. These will be discussed.

11:00am **AS+BI+VT-TuM10 Mass spectrometry surface analysis outside the vacuum**, *Justin Wiseman, M.E. ElNaggar, J.K. Kennedy, B.L. Laughlin*, Prosofia Inc. **INVITED**

Advances in mass spectrometry in the last 20 years has produced instruments with higher resolving power, smaller footprints, even portable, and the capability of measuring surfaces for molecules in the ambient air; the former truly enabling the latter. Ambient mass spectrometry involves the characterization of samples in their native state in the open air and is exemplified by the development of Desorption Electrospray Ionization (DESI) and Direct Analysis in Real Time (DART). DESI uses high velocity charged droplets produced by a pneumatically-assisted electrospray to effect desorption and ionization of surface-bearing analytes. The applications of the technique are broad and span from the detection of leachables to thin-layer chromatography to imaging of drugs, metabolites and lipids in histological tissue sections, where the lateral spatial resolution has been reported to be as high as 50 $\mu$ m. The flowprobe, also an ambient technique, uses a liquid-microjunction formed at the surface to extract and deliver analytes to the mass spectrometer via an electrospray source. The applications of the flowprobe are also broad and have included microarray sampling, thin-layer chromatography plate analysis, and biological tissue analysis. This presentation will discuss the merits and applications of each of the DESI and flowprobe devices, with emphasis on their application to imaging biological tissue.

11:40am **AS+BI+VT-TuM12 Transporting Ions from Ambient Pressure into Vacuum for Lab-based and Mobile Mass Spectrometers**, *Mitch Wells*, FLIR Mass Spectrometry **INVITED**

The proliferation of Atmospheric Pressure Ionization (API) sources for mass spectrometry (MS) has expanded the applicability of the MS analysis technique to a wide range of chemical and biological challenges, to the extent that the 2002 Nobel Prize in Chemistry was awarded to John Fenn and Koichi Tanaka for their development of Electrospray Ionization (ESI) and Matrix-assisted Laser Desorption Ionization (MALDI), respectively. Furthermore, recent developments in a specific category of API, referred to as Ambient Ionization (AI), have simplified the applicability of API techniques by removing some or all of the need for sample preparation prior to analysis. AI techniques, such as Desorption Electrospray Ionization (DESI), Direct Analysis in Real Time (DART), and an ever increasing list of additional techniques and variations, allow for direct analysis of an enormous range of sample and matrix types; whole blood, illicit drugs in fingerprints, tissue cross-sections, pharmaceuticals, and forensic samples have all been examined with AI, to name just a relatively few examples.

All API techniques have in common the need to transport ions from atmospheric pressure into the high vacuum of the mass spectrometer - typically  $<10^{-5}$  Torr ( $<1$  mPa). Various ion sampling and transport mechanisms are used to transfer ions through differentially-pumped vacuum stages to the mass analyzer. In all cases, significant losses at each stage mean that only a very small fraction ( $<<1\%$ ) of the ions generated from a sample are actually analyzed. The situation is even worse for systems that are intended to be used in mobile or field labs, where space and power are at a premium and large pumping systems are therefore not acceptable.

This talk will briefly review AI techniques to illustrate their value in analytical chemistry (including biological, clinical, and forensic analysis), and will then describe means by which ions are transported from atmosphere into vacuum, with the hope of stimulating dialog with the vacuum community about ways and means that this process could be improved, especially for small, rugged instruments designed for outside-the-lab use.

## Vacuum Technology

Room: 303 - Session VT-TuM

## Gas Dynamics, Modeling, and Pumping Systems

**Moderator:** Lily Wang, Los Alamos National Laboratory, Martin Wüest, INFICON Ltd., Liechtenstein

8:00am **VT-TuM1 A Fast Numerical Method for Determining the Pressure Distribution in Electrostatic Chucks**, *Jack McNerney*, Lam Research Corp

The excellent cooling capabilities of the electrostatic chuck enable high power plasma etching of modern semiconductor devices. In order to maintain a uniform temperature across the silicon wafer, a thin layer of helium is inserted between the wafer and chuck. Some of this gas leaks out at the wafer edge, and the resulting flow of helium can lead to pressure drops that compromise the heat transfer uniformity of the chuck. Fluid

dynamics modeling of the helium distribution is often used in the design phase to ensure uniform pressure under various scenarios.

Because of the small gaps and low pressures, the gas behind the wafer is in the transitional or molecular flow regime. Modeling electrostatic chuck designs then requires using very computationally expensive methods such as Direct Simulation Monte Carlo (DSMC). In this paper, a simplified modeling approach is developed that allows the pressure distribution to be modeled as a two-dimensional conductance problem. This is done by extending the conductance calculations used in one-dimensional vacuum piping networks. The accuracy of the method is compared to molecular flow modeling. The method is then used to model some chuck configurations.

8:20am **VT-TuM2 Numerical Simulation of a Jet Disrupter in an Electrospray RF Ion Funnel**, *Eric Tridas, R. Schlaf*, University of South Florida, *M. Anthony*, Elion Systems

Electrospray ionization (ESI) is a versatile method for creating gas phase ions from solution while maintaining the native chemical functionality of the solute. Using this method, functional bio- and macromolecular thin films can be produced for use in biosensors, scaffolding for tissue generation, photovoltaics and other emerging fields of research. The Macromolecular Patterning System, designed and constructed at the University of South Florida (USF), utilizes ESI as a material source to create such films. The system is comprised of three differential pumping stages, each containing custom designed electrodes used to define the trajectory of the ions. The focus of this study is on the first of the three stages which contains a radio frequency (RF) ion funnel. Computational fluid dynamics (CFD) simulations of the air flow into this chamber were performed and coupled with simulations calculating the generated electric field. Using the ion trajectory simulation software SIMION, the flight paths of ions within this first chamber were calculated. Experiments were then performed to test the results of the simulations. A "randomization parameter" based on the turbulence kinetic energy of the CFD simulations was used to model the time-varying component of the flow velocity yielding a result that closely matched the system. Variation of electrode voltages in the physical apparatus yielded similar results to those obtained from the simulations. Most significantly, the overall trend and peak values of ion transmission were accurately predicted from the simulations.

8:40am **VT-TuM3 Gas Dynamics Modelling Efforts at CERN**, *Roberto Kersevan*, CERN, Switzerland **INVITED**

The Vacuum Surfaces and Coatings (VSC) Group at CERN is involved in several large projects, either at CERN or in collaboration and/or support of other laboratories. The design of new vacuum chambers and components is taking a considerable amount of time of many physicist and engineers in the VSC. New accelerators are being designed, either for fabrication and installation in a short time or as part of the European strategy for future accelerators. Two extreme examples are: 1) The ELENA project (Extra-Low Energy Accelerator ring) a  $\sim 30$  m circumference 100 keV anti-proton decelerator aimed at increasing the anti-proton production from the existing AD machine (Antiproton Decelerator ring). ELENA will require an average pressure better than  $3E-12$  Torr. ELENA is under design and construction now, with expected start of commissioning in 2016. On the other side of the spectrum are the TLEP and HE-LHC machines, which are part of the Future Circular Colliders lepton-lepton and hadron-hadron (FCC-ee, FCC-hh) versions of colliders aiming at greatly improving the production of Higgs and  $W/Z$  bosons, and top quarks (FCC-ee), and raising the center-of-mass energy in the 50+50 TeV range (FCC-hh). Such conceptual machines would require circumferences in an unprecedented 80~100+ km range. The FCC-ee versions would generate of the order of 50 MW of synchrotron radiation (SR) for 175+175 GeV electron-positron beams, and the FCC-hh would generate as well a considerable amount of SR in the 4~5 keV critical energy range, thanks to 16-20 tesla superconducting magnets. The already approved High-Luminosity upgrade of the LHC (HL-LHC) and this FCC program will require a thorough upgrade of the injector chain, composed of some accelerators which are today exceeding the 50 year mark. In parallel, there are accelerators like the HIE-ISOLDE upgrade of the ISOLDE machine, aimed at post-acceleration of radioactive ion-beams, and new-concept experiments like the AWAKE plasma-acceleration project. In order to tackle all these projects, the VSC group has decided to develop several numerical analysis tools, namely test-particle monte-carlo (TPMC) codes and the electrical-network analogy (ENA) (implemented via the Ltspace freeware). This paper will briefly describe the various codes developed (Molflow+ for molecular flow, SYNRAD+ for SR, and McCRYO-T for radiative heat exchange) and the ENA approach. It will then show some examples of the application of these codes to CERN projects and also comparison and benchmarking with results published in the gas dynamics field and dedicated experiments carried out at CERN.

9:20am **VT-TuM5 Mixture Flow of Rarefied Gases through a Thin Orifice Over the Whole Range of Gas Rarefaction, Felix Sharipov**, Federal University of Parana, Brazil

Rarefied gas flow through a thin orifice is well studied on the basis of the direct simulation Monte Carlo (DSMC) method, see e.g. Ref.[1,2]. In spite of the fact that in practice one deals with gaseous mixtures more often than with a single gas, the information about such kind of flows is still poor. That is why it is attractive to treat gaseous mixtures as a single gas with the molecular mass equal to its average values of the corresponding mixture. However, such an approach not always provides reliable results. The aim of the present work is a numerical modeling of mixture flows of rarefied gases through a thin orifice on the basis of the direct simulation Monte Carlo (DSMC) method. The mass flow rate and flow field are calculated over the whole range of the rarefaction parameter for various values of the pressure ratio and for several values of the mole fraction. A comparison of the present numerical results with those obtained for a single gas is performed. A recommendation of the applicability of single gas results to gaseous mixture is given.

#### References

1. F. Sharipov, Numerical simulation of rarefied gas flow through a thin orifice. *J. Fluid Mech.* Vol.518, pp. 35-60 (2004).
2. F. Sharipov and J.L. Strapasson, Ab initio simulation of rarefied gas flow through a thin orifice. accepted in *Vacuum*.

9:40am **VT-TuM6 Numerical Modeling of Particle Transport in Rarefied Flow, Andreas Mack, Van der Donck, O. Kievit**, TNO Delft, the Netherlands

Within wafer handling devices, environments from ambient pressure to ultra-high vacuum are present. The wafers are moved by robots between the compartments which are separated by load locks. With closed load lock valves, the pressure is reduced by pumping-down such that the pressure level of the next compartment is roughly matched. Since the pumping speed is approximately constant, the pumping time to very low pressures would take long or require additional pumps such that usually the target pressure is only matched by two orders of magnitude. The final pressure is then achieved by opening the load lock valve such that the pressure in both compartments reach equilibrium. This process includes a strong expansion of the flow such that locally very high flow velocities can be reached up to supersonic speed. Since the flow is in the rarefied regime, the forces on surfaces such as the wafer are small but particles released during the wafer handling process can be dispersed downstream due to drag or gravitational forces. Since there is only sparse information available about the coupling between contaminating particles and the rarefied flow, the present paper focusses on the numerical modelling of particle transport in rarefied flow. A DSMC (Direct Simulation Monte Carlo) code is applied to typical domains of wafer handling such as load lock valves and coupled with a particle tracer. Both codes are available within the open source software package OpenFoam and have been validated in the relevant regimes by either generic numerical experiments or, where available, experimental or other numerical data. By this, particle contamination in low pressure environments can be investigated. On the one hand, possible particle contamination regions and active or passive measures to reduce particle contamination can be identified. On the other hand, the global dispersion behavior of different particle classes is investigated such that conclusions over the generic movement of particles within a low pressure environment can be drawn. By this, the dispersion of certain particles can be excluded due to geometrical or physical constraints which is valuable information for particle contamination measurement. Beside the modelling of particle transport for the generic valve opening between compartments the present paper includes also the venting-up of a representative load lock to ambient pressure whereas the results of pseudo-3d and a full 3D modelling are discussed with respect to flow topology and particle contamination.

Keywords: DSMC, rarefied flow, particle contamination, load lock

11:00am **VT-TuM10 Improving the Performances of Getter Pumps: Recent Developments in NEG Technology, Fabrizio Siviero, G. Bongiorno, L. Caruso, A. Gallitognotta, L. Viale, E. Maccallini, P. Manini**, SAES Getters, Italy

**INVITED**

Non Evaporable Getter (NEG) pumps are commonly used when large pumping speeds for H<sub>2</sub> and active gases (i.e., H<sub>2</sub>O, O<sub>2</sub>, CO, CO<sub>2</sub>) are required. The NEG pumps are very small, lightweight, with reduced magnetic interference, cause no vibration, and consume negligible power. Thanks to these qualities NEG pumps are widespread in many UHV applications.

Nevertheless, some factors still limit an even wider diffusion of NEG pump technology, mainly related to the topics of particle release and gas evolution during activation. Indeed, as a precaution, NEG pumps are generally not used in application requiring particle-free environment. Only recently,

measurements performed at a large accelerator facility have shown the compatibility of St172<sup>®</sup> alloy sintered getters in particle free environments, i.e RF cavities, after suitable treatments. Also, it is well known during getter activation hydrogen as well as other physisorbed species are desorbed causing pressure increase up to the 1e-6 mbar range. In some applications this is seen as a problem due, for example to constraints in pumping efficiently hydrogen away from long and narrow chambers.

These topics will be discussed based on recent outcomes of research activities carried out in SAES R&D labs to address these issues. New alloys, belonging to the ZAO<sup>®</sup> family, and new production processes have been developed, showing interesting characteristics. Among them we highlight intrinsically reduced particle release, lower hydrogen equilibrium isotherms and more efficient management of the gas load during activation. These properties are combined with a new pump design, even more compact and simpler to install than before, with the aim of providing an enlarged community of users with smarter solutions to their vacuum needs.

11:40am **VT-TuM12 Advanced High Speed Water Vapor Cryopumps: Enabling Today's Vacuum Processes, Kevin Flynn, C. Rebecchi**, Brooks Automation, Inc., Polycold

**INVITED**

Effective and efficient pumping of water vapor in vacuum systems via cryopumps or cryotraps is critical to achieving required vacuum system performance for many processes. A brief overview of general cryopumping methods is presented along with an in depth review of water vapor cryopumping. Water is of special importance due to its difficulty to be pumped, its deleterious impact on coatings when it remains on the substrate, and its ability to form oxygen which reacts with and degrades thin film quality. In addition to the basic pumping function, the ability of a cryopump to rapidly cool and defrost or regenerate the cryosurface are important for enabling high chamber productivity. Required water vapor partial pressures vary widely among typical vacuum processes, ranging from 10<sup>-1</sup> torr to below 10<sup>-9</sup> torr. Similarly required pumping speeds range from thousands of liters per second to over 200,000 liters per second. The combination of these varying requirements drive different demands for cryopump cooling capacity and temperatures. A variety of different vacuum applications covering medium, high and ultra high vacuum applications, and including batch and inline processes, are reviewed along with considerations of cryosurface location relative to the chamber and process.

# Tuesday Afternoon, November 11, 2014

## Vacuum Technology

Room: 303 - Session VT-TuA

### Vacuum Quality Analysis, Outgassing, and Control

**Moderator:** James Fedchak, National Institute of Standards and Technology (NIST), Marcy Stutzman, Thomas Jefferson National Accelerator Facility

2:20pm **VT-TuA1 Our Present Understanding of Outgassing, Manfred Leisch**, Graz University of Tech., Austria **INVITED**

Outgassing means basically the diffusion of atoms usually hydrogen through the bulk material, entering the surface and desorbing from it. The important consequence is it limits the lowest achievable pressure in a vacuum chamber and is a central issue in vacuum science with respect to ultra high (UHV) and extreme high vacuum (XHV). Stainless steel (SS) is one of the most commonly used constructional materials for vacuum chambers and components. A considerable body of work is documented on the hydrogen outgassing behaviour of SS. For the description of the outgassing rate basically two models common as diffusion limited model (DLM) and recombination limited model (RLM) have been discussed so far. Experimental studies in the last decade show that the real situation on the complex SS surface cannot be fully described by DLM or RLM. Hydrogen atoms approaching the surface from the bulk are desorbing in a second-order process. The rate of recombination depends strongly on the atomic structure of the surface and is e.g. generally higher on stepped surfaces than on flat close packed planes. A new insight was gained by atomic level studies on the real morphology of SS with atomic force microscopy (AFM) and the scanning tunnelling microscopy (STM).

Beside surface morphology surface composition additionally controls the desorption kinetics. Auger electron spectroscopy (AES) gives reason for a composition change. Since the information depth of AES covers several atomic layers complementary atom probe analysis were performed, measuring the chemical composition on the surface atomic layer by layer. Energy calculations using the ASED method (Atom superposition and Electron Delocalization) result in lower energy levels in Fe vacancies. It supports the picture that surface and subsurface defects form traps with different energetic levels. They may control the recombinative desorption process and give explanation for the observed outgassing behaviour of stainless steel. From this results a more complete description of the outgassing process may be given by a more or less dynamic equilibrium between diffusion, sojourn in different level traps and recombinative desorption.

This work was supported by the Austrian „Fonds zur Förderung der wissenschaftlichen Forschung“ P 12099 and “Zukunftsfonds Steiermark” P 119.

3:00pm **VT-TuA3 Hydrogen Traps in the Outgassing Model of a Stainless Steel Vacuum Chamber, Robert Berg**, National Institute of Standards and Technology (NIST)

The outgassing model accounts for the geometry of the chamber components, the hydrogen dissolved in those components, and the processes of diffusion, recombination, and trapping. Strongly bound or “trapped” hydrogen, which occurs at heterogeneities such as dislocations and grain boundaries, can hold most of the dissolved hydrogen even though those locations comprise fewer than 0.1% of all lattice sites. Four simplifications allowed practical use of the model: (1) Each component was described as a one-dimensional object. (2) The hydrogen initially dissolved in each component was described as a uniform concentration. (3) Accurate, consistent values were used to describe diffusion and recombination in stainless steel types 304 and 316 [Grant et al., J. Nucl. Mater. 149, 180 (1987); 152, 139 (1988)]. (4) Only one type of hydrogen trap was considered, and trapping was ignored in components made from vacuum remelted stainless steel. The simple model was developed and validated by comparing it to outgassing measurements. Traps were required to describe the outgassing from a component made of drawn stainless steel 304. The initial hydrogen concentration in that component was comparable to concentrations found elsewhere by thermal desorption and almost 100 times larger than in the components made of vacuum remelted 316 stainless steel. The model’s usefulness is illustrated by using it to predict the outgassing of a vacuum chamber made of type 304 stainless steel.

3:20pm **VT-TuA4 A Mild Steel Ultrahigh Vacuum Chamber Appropriate for Magnetic Shielding, B. Cho, S.J. Ahn**, Korea Research Institute of Standards and Science (KRISS), Republic of Korea, *C.D. Park, Taekyun Ha*, POSTECH, Republic of Korea

Mild steel, i.e. low carbon steel, is a soft magnetic material and widely used for shielding sensitive experimental apparatuses from stray magnetic field because of its relatively low price and high magnetic permeability. Mild steel vacuum chambers are usually nickel-plated in order to prevent corrosion and improve the vacuum. For example, electron microscopes employ nickel plated mild steel for constructing their specimen vacuum chambers in which the electron beam propagates and interacts with specimens; presence of stray magnetic field deteriorates proper propagation of the electron beam, degrading the resolution of the electron microscope.

The mild steel has not been employed, to the best of authors’ knowledge, for ultra-high vacuum (UHV) use because its outgassing rate has been known to be too high; reported values were on the order of  $10^{-8}$ – $10^{-9}$  mbar  $l s^{-1} cm^{-2}$  or higher [1]. Ishimori *et al.* [2] reported that the outgassing rates of a mild steel (carbon ~0.15%), a chromium-plated mild steel and a stainless steel were  $2\sim 3 \times 10^{-11}$  mbar  $l s^{-1} cm^{-2}$ ,  $7\sim 9 \times 10^{-11}$  mbar  $l s^{-1} cm^{-2}$  and  $2\sim 3 \times 10^{-12}$  mbar  $l s^{-1} cm^{-2}$ , respectively, after baking at 300 °C for 3 hours. The outgassing rate of UHV chambers are normally on the order of  $10^{-12}$  mbar  $l s^{-1} cm^{-2}$  or less after baking at 100 ~ 200 °C.

The outgassing rates of a mild steel and a stainless steel 304 chamber were measured by using the so-called rate-of-rise (RoR) method [3]. We present that the outgassing rate of the mild steel purchasable on the market is much smaller than that of a stainless steel type 304L which is most widely used as a UHV vacuum chamber material. The ultimate pressure of a vacuum chamber made of the mild steel was  $2.7 \times 10^{-11}$  mbar, and its outgassing rate was of  $< 3 \times 10^{-14}$  mbar  $l s^{-1} cm^{-2}$ , which indicates the mild steel is even appropriate for extreme high vacuum use. Vacuum annealing of the mild steel at 850 °C reduced the outgassing rate further.

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[2] Yoshio Ishimori, Nagamitsu Yoshimura, Shuzo Hasegawa, and Hisashi Oikawa, SHINKU 14(8), 295(1971).

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4:20pm **VT-TuA7 Ultimate Limits in the Gas Composition Determination Within Small Sealed Volumes by Quadrupole Mass Spectrometry, Vincenc Nemanic**, Jozef Stefan Institute, Slovenia **INVITED**

Miniaturization of modern sealed vacuum devices and higher demands for their stable operation on the long-term scale require accurate determination of the gas composition in the early stage of their operation, as well as after a long operating period. Since particular gases may have detrimental effect on the device performance even at low concentrations, accurate quantification of the gas mixture is as important as well as a challenging task. Among a few highly gas-sensitive methods capable to detect quantities below  $10^{-4}$  mbar L, the quadrupole mass spectrometry seems to be the most appropriate one for this task.

A two-step procedure, consisting of sample puncture inside an expanding chamber, followed by opening the leak valve to the quadrupole mass spectrometer, kept in the analytical chamber at  $\sim 3 \times 10^{-11}$  mbar, is proposed. A limited number of ion current readings are used for the reconstruction of the original total pressure and gas composition. Calibration of such instruments at particular partial pressure is regularly achieved at stable gas influx and constant pumping speed. Several discrete points have to be recorded to get the sensitivity of the instrument expressed in A/mbar.

In this presentation, a systematic approach for preparing the instrument for routine quantification of small gas amounts is described. In the first stage, the instrument was calibrated as the precise partial gas flow meter by an innovative *in-situ* calibration procedure by three different gases, hydrogen, argon and nitrogen. Each gas was admitted into the expanding chamber, having a precisely determined volume of 0.312 L and equipped by a capacitance manometer. By opening the leak valve, ion currents versus gas flux were recorded over three orders of magnitude, expressing the partial flux sensitivity in As/(mbar L). In the second stage, known gas quantities  $\sim 10^{-4}$  mbar L of pure gas were admitted at different leak valve conductance to determine the instrument’s response. This data enabled minimizing the error by searching for a compromise between the number of the readings and the level of recorded ion currents. In the third stage, gas mixtures with various contents of three gases were prepared and analyzed. This evaluation enabled a much better prediction of the ultimate limits in reconstructing of the unknown gas mixture in a real device. Anyhow, uncertainty in



evaluation increases by lowering the gas amounts as ion currents become indistinguishable from the background readings of the instrument.

5:00pm **VT-TuA9 The Importance of Competitive Langmuir Adsorption Kinetics for Vacuum Cleanliness**, *Richard Versluis*, TNO Technical Sciences, Netherlands

There are numerous examples of systems that rely on a very clean vacuum. Gaseous contamination and surface contamination may influence the process or even damage the machine (such as beam scattering accelerators and e-beam equipment or background contamination in gas analyzers) or may contaminate samples (XPS, SEM, HIM etc) or may contaminate sub-components of the machine (such as mirror contamination in EUV systems, space systems, spectral analyzers etc). The usual methods of contamination inspection are measurement of the residual gas compositions (RGA's) or the use of witness plates to determine surface contamination. The requirements on residual contamination (either gaseous or adsorbed on surfaces) are becoming more stringent with the development of equipment that is becoming more sensitive for contamination. A good understanding of the kinetics of contamination transport and gas-surface interaction is crucial when developing or using these requirements. This is important for both equipment users and equipment developers.

This talk will highlight some important aspects of gas-surface interaction in vacuum chambers by focusing on the dynamical behavior of gas species competing for adsorption sites. We will show how the adsorption energies and concentrations influence the equilibrium that is reached, but we will also show how the adsorption energies and the concentrations determine the kinetic behavior before equilibrium is reached and how they influence surface coverage by different species during non-equilibrium. A simple model solving the coupled kinetic equations is able to predict the time dependent behavior, which can be used to determine for instance outgassing times and sampling times and the relationship between measured gas concentrations and surface coverage during non-equilibrium.

5:20pm **VT-TuA10 Diagnostic Tool to Identify Volatile Molecules in Vacuum**, *Freek Molkenboer, A. Van de Runstraat, J.A. Van der Meer, T. Van Groningen, O. Kievit*, TNO Technical Sciences, Netherlands

Residual gas analyzers (RGAs) are commonly used in ultra-high vacuum applications to measure vacuum quality. The RGA fragments and ionizes the molecules that are present in the gas phase in the vacuum system. These fragments of all the molecules make up the RGA spectrum. The RGA spectrum has to be interpreted to identify the contaminants that are present in the vacuum system. This is complicated and often impossible in case of complex mixtures of organics in the vacuum atmosphere.

The goal of this project is to develop a simple-to-use diagnostic tool that is able to identify the contaminant molecules in vacuum directly. After a trade-off of various options, we selected a removable cold trap in combination with an off-line gas chromatography-mass spectrometry (GC-MS) system for analysis of the samples.

The cold trap is installed on a vacuum flange. A removable sample tube is positioned inside the cold trap in connection with the vacuum system. The cooling of the cold trap is achieved with Peltier elements, which makes it simple to operate as well as independent of supply of coolants such as liquid nitrogen. After sampling, the sample tube can be removed without venting the vacuum system and a new sample tube can be installed to continue measurements if required.

After sampling of the vacuum vessel, the sample tube is connected to a GC-MS system for analysis of the sample and identifying and partly quantify the organic molecules present.

The first results are promising and we continue to improve the system. In this presentation we will present our sampling method and the results of vacuum quality measurements using the new diagnostic tool.

5:40pm **VT-TuA11 Quantitative Gas Analysis of Small Batch Samples by Quadrupole Mass Spectrometer**, *Lily Wang*, Los Alamos National Laboratory

In our studies of static gas release properties of various solid materials at low temperatures ranging from 25 to 80 °C, we find the amounts of gas collected from the experimental samples in sealed vacuum vessels over extended times (weeks to months) are only a few to less than 100 torrs in a free volume of 10 - 50 cc. In order to analyze these small batch gas samples, a quantitative method was developed using a quadrupole mass spectrometer. This method involves introducing a small pulse of the gas with a custom-designed sample manifold into a quadrupole mass spectrometer and analyzing the gas component quantity in a few seconds. The method is relatively quick and is particularly suitable for gas components that have low sticking coefficients to stainless steel surfaces. This method was evaluated for hydrogen, methane, and argon. In this

presentation, the setup, the calibration and measurement procedures, and the performance of the method are presented and discussed.

6:00pm **VT-TuA12 A Novel Vacuum Mini-Environment Design For Thin Film Sputter Deposition Apparatus**, *Jun Xie, R.L. Ruck, C. Liu, P. Leahey, T. Bluck*, Intevac, Inc.

In microelectronics manufacturing, many critical process steps are carried out in high vacuum apparatus. Trace quantities of residual gaseous species, such as hydrogen and water (H<sub>2</sub>O), are always present within such system, which are of great concern to state-of-the-art device fabrication. In the hard-disk drive industry, for example, metallic thin films of a magnetic recording disk are especially susceptible to H<sub>2</sub>O, which could affect film growth adversely and compromise the device performance. It becomes a top priority to prevent the trace contaminants in the vacuum system, especially H<sub>2</sub>O, from interacting with these metallic thin films during a sputter deposition process. The goal of this study is to create a pristinely clean mini-environment inside a general vacuum apparatus for ultrapure thin film sputter deposition. The approach consists of two innovative features. The first is a retractable enclosure that seals off a volume around the sputter target and the substrate. The second is a series of pumping channels of pre-determined sizes and shapes through the wall of the enclosure that facilitate the evacuation of the gases or byproducts from the enclosure in a controlled manner while minimizing the probability of outside contaminants entering the enclosure. Experiments were conducted by sputter-depositing chromium thin films in such an enclosure to getter contaminants and assess its effectiveness with secondary ion mass spectrometry (SIMS) and X-ray photoelectron spectroscopy (XPS) analysis.

# Tuesday Evening Poster Sessions

## Vacuum Technology

Room: Hall D - Session VT-TuP

### Vacuum Technology Division Poster Session and Student Poster Contest

**VT-TuP2 Performance Evaluation of Scroll Pump.** *Fan-Chun Hsieh, P.H. Lin, C.S. Yu, F.Z. Chen*, National Applied Research Laboratories, Taiwan

Scroll pumps are widely used in solar-optic and semiconductor industry for backing purpose. The performance of scroll pump could affect the operation of production line significantly. Here, we compared the performance of scroll pump before and after maintenance. The ultimate pressure and acceleration were measured. The ultimate pressure decreases slightly from  $5 \times 10^{-1}$  Torr to  $2 \times 10^{-2}$  Torr. The RMS magnitude of acceleration shows a maximum peak which occurs at about 30 Hz. Moreover, small peaks were also observed in the spectrum. The small peaks were high before maintenance compared to after maintenance. We speculated that this trend is due to the damage of ball bearing and wear of tip seal. The proposed measurements provide a considerable advancement in maintenance of pump.

**VT-TuP3 Reliability Engineering Study of TMPs and Cryopumps.** *JongYeon Lim, K.M. Choi, K.M. Baik*, Korea Research Institute of Standards and Science, Republic of Korea, *S.Y. In*, Korea Atomic Energy Research Institute, Republic of Korea, *S.K. Lim*, National Nano Fab Center, Republic of Korea, *D.Y. Koh*, Korea Institute of Machinery and Materials, Republic of Korea, *W.S. Cheung*, Korea Research Institute of Standards and Science, Republic of Korea

Methods of the characteristics evaluation of vacuum pumps are well-defined in the international measurement standards such as ISO, PNEUROP, DIN, JIS, and AVS. HV pumps such as TMPs and cryo-pumps, essential equipment in the advanced industry, require very objective engineering reliability for ascertaining their performance estimation.

Developmental effort for establishing reliability engineering evaluation infra-structure of TMPs and cryo-pumps has been conducted in the manner of component level test, characteristics evaluation and field tests in KRIS.

Until now reliability study for vacuum pumps is not well developed since there are so many unknown factors are prevailing in vacuum industry. However, the increasing needs for the reliability engineering in real field are continuously rising. In this reason we have currently developed the partial reliability engineering evaluation system of high vacuum pumps. In the case of TMPs and cryo-pumps, we shortly try to remark the developmental flow of reliability chain in the fields as follows;

TMPs:

1. Characteristics evaluation – pumping speed, ultimate pressure, etc.
2. Endurance evaluation – Continuous starting and stopping operation (10 % to 90 % RPM)
3. Field Test – NNFC Etcher system
4. Quality assurance evaluation – magnetic field tolerance valuation system
5. Destructive test,

Cryo-pumps:

1. Component evaluation – Refrigerator evaluation
2. Characteristics evaluation – pumping speed, ultimate pressure, etc.
3. Field Test – NNFC Sputter system
4. Quality assurance evaluation .

In this presentation we suggest the methodological approach to the completion of high vacuum pump development through a reliability engineering study

*Acknowledgements: Results are partially attributed to two national project (Contract No. 10031836) sponsored by the Korean Ministry of Knowledge Economy, and KRIS main project 14011016.*

**VT-TuP4 Improved Threshold Ionisation Mass Spectrometry.** *D.L. Seymour, S. Davies, Alan Rees, P. Hatton*, Hiden Analytical, UK

Threshold ionisation mass spectrometry (TIMS) is well established as a technique for improving on standard methods of residual gas analysis, particularly when the dominant peaks in the mass spectrum occur at very nearly the same mass/charge ratio. The mass peaks are then difficult to separate using standard quadrupole mass spectrometric methods. We have

described elsewhere several applications in which TIMS has proved to be very effective. More recently, we have examined ways in which the interpretation of TIMS measurements can be improved by fitting to the experimental data trend lines calculated from theoretical expressions.

**VT-TuP5 The XHV Cathode Preparation System of the "High Current High Polarization" Electron Gun for the Proposed eRHIC Project.** *Omer Rahman, I. Ben-Zvi, E. Wang, T. Rao, J. Skaritka*, Brookhaven National Laboratory

A very compact cathode preparation chamber for the high current high polarization gun for the proposed Erhic project has been designed and assembled at the Brookhaven National Laboratory. This preparation chamber is used to prepare GaAs cathodes to be used to extract electron beam in the multi-cathode gun. Preparation of GaAs strictly requires XHV environment and this system is able to achieve that in a consistent way. In this paper, the construction of the vacuum system including different components, the procedure and pressure results over more than a year of study will be discussed.

# Wednesday Morning, November 12, 2014

## Vacuum Technology

Room: 303 - Session VT-WeM

### Accelerator and Large Vacuum Systems I

**Moderator:** Marcy Stutzman, Thomas Jefferson National Accelerator Facility

8:00am **VT-WeM1 Vacuum Technology Developments at Daresbury Laboratory for Modern Accelerators, Keith Middleman, A.N. Hannah, J.D. Herbert, O.B. Malyshev, R. Valizadeh, STFC Daresbury Laboratory, UK** **INVITED**

The Vacuum Science group at the STFC Daresbury Laboratory has a unique position in that it has the capability to operate and design the vacuum systems for new accelerators whilst maintaining a very active research laboratory looking at many new facets of vacuum design for accelerators. This gives the group the opportunity to develop ideas in the laboratory before implementing them on the accelerator. This paper will present some of the latest accelerator ideas and machines at Daresbury and provide an insight into how some of our laboratory developments are helping improve the vacuum design.

A range of topics will be covered such as:

- 1) Machine developments – VELA, CLARA and ALICE
- 2) NEG coatings – a new quaternary alloy with a reduced activation temperature
- 3) Photocathode research – metal and semiconductor cathode developments
- 4) Bakeout – a new permanent thin film heater coating for in-situ bakeout
- 5) XHV – optimising the process to routinely achieve  $10^{-12}$  mbar
- 6) Thin films – SRF coating developments

8:40am **VT-WeM3 First Year Operation of NSLS-II Vacuum Systems with Beam, Hsiao-Chuan Hseuh, W. DeBoer, S. DiStefano, C. Hetzel, S. Leng, K. Wilson, D. Zigrosser, H. Xu, Brookhaven National Laboratory**

National Synchrotron Light Source II is a new synchrotron radiation facility, consisting of a 200-MeV Linac, a 3-GeV Booster and a 3-GeV storage ring. The Linac and the Booster were completed and commissioned in 2012 and 2013, respectively. The 792m storage ring was completed in January. Commissioning with electron beam is underway in the last few months. The performance of these ultrahigh vacuum systems with intense electron and X-ray beams will be described. The reliability and usefulness of over 1500 vacuum pumps and instruments will be summarized. Experience with NEG coated narrow gap chambers, in-vacuum undulators and superconducting cavity will also be presented.

\*Work performed under the auspices of U.S. Department of Energy, under contract DE-AC02-98CH10886.

9:00am **VT-WeM4 APS-Upgrade Storage Ring Vacuum System Conceptual Design, Herman Cease, B. Stillwell, B. Brajuskovic, J. Nudell, J. Carter, Argonne National Laboratory**

A conceptual design is being developed for a storage ring vacuum system at the Advanced Photon Source (APS) which is compatible with a seven-bend achromat lattice under consideration by the APS Upgrade (APS-U) project. The required proximity of the magnet poles to the beam, quantity and stability of beam position monitors, synchrotron radiation loading, beam physics considerations, and installation duration place a challenging set of constraints on the vacuum system design. These requirements can be satisfied with a hybrid system which combines NEG-coated copper chambers with conventional extruded aluminum chambers housing mechanically-mounted NEG strips and discrete absorbers. The hybrid design, expected outgassing and thermal loads, and preliminary vacuum pressure analysis is described.

9:20am **VT-WeM5 APS-Upgrade Vacuum System Pressure Using a 3-D Simulation Tool, Jason Carter, H. Cease, Argonne National Laboratory**

A conceptual design is being developed for a storage ring vacuum system at the Advanced Photon Source (APS) which is compatible with a seven-bend achromat lattice under consideration by the APS Upgrade (APS-U) project. The design features a hybrid vacuum system to address outgassing and thermal loads which will combine discrete pumping, NEG-coated copper chambers, and NEG strip pumping in conventional extruded aluminum chambers. A preliminary 3D vacuum pressure analysis is described which uses the SynRad/MolFlow+ 3D vacuum analysis package developed at CERN to determine if the system can meet pressure requirements. The

analysis uses the software package to predict photon stimulated desorption loads and then determine the resulting vacuum system pressure.

9:40am **VT-WeM6 For Some Results Solving Key Issues of Vacuum Systems in Electron Storage Rings, Hiroshi Saeki, Japan Synchrotron Radiation Research Institute, Japan, T. Momose, Emeritus Professor of Miyagi National College of Technology, Japan**

From 1980's to 2000's in Japan, electron storage rings had some key issues of their vacuum systems. To overcome two key issues especially, we observed dust trapping phenomena as a key issue, using lead-glass counters at first.<sup>1,2</sup> Dust particles collected in beam ducts were analyzed<sup>3</sup> and simulation experiments to trap dust particles were carried out using these collected dust particles.<sup>2,4,5</sup> As the results, the mechanism of the phenomena was found clearly and we knew how to prevent these phenomena occurring.<sup>6,7</sup> We review and discuss the phenomena using published and unpublished data.

As the other key issue, it was that ionization gauges misread due to radiation-induced currents.<sup>8</sup> A hot-cathode-ionization gauge with correcting electrode was developed and tested in simulation experiments<sup>9,10,11</sup> and in actual radiation environments.<sup>12,13</sup> The pressure-measurement error of the developed vacuum gauge was about 20%. We also review and discuss the vacuum gauge and other devices<sup>14</sup> to overcome other issues using latest data.

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11:00am **VT-WeM10 Progress in Thin Film Technology for Superconducting RF Applications, Anne-Marie Valente-Feliciano, Thomas Jefferson National Accelerator Facility** **INVITED**

Bulk Nb has so far been the material of choice for Superconducting RF (SRF) applications. With RF cavity performance approaching the theoretical limit for bulk niobium, alternative routes for the future of superconducting structures used in accelerators are explored. Over the years, Nb/Cu technology has positioned itself as an alternative route for the future of superconducting structures used in accelerators, but has suffered shortcomings due to the commonly used magnetron sputtering. The recent developments in ionized PVD coating techniques (i.e. vacuum deposition techniques using energetic ions) such as Electron Cyclotron Resonance (ECR) and High Power Impulse Magnetron Sputtering (HiPIMS) are opening avenues for the production of thin films tailored for SRF applications based on Nb and alternative materials.

This contribution reports the on-going efforts pursued at Jefferson Lab and in different institutions to exploit the potential of novel film technologies to produce bulk-like Nb films and go beyond Nb performance with the development of film systems, based on other superconducting materials and multilayer structures.

11:40am **VT-WeM12 Modeling and Measurement of a Tesla-like Cage Cavity**, *John Noonan, M.J. Virgo, T.L. Smith*, Argonne National Laboratory

The cage cavity is an RF cavity fabricated by forming tubes to follow the surface contour of a cavity design, e.g. a TESLA cavity, and assembling the tubes to form a closed cavity. Computer simulations demonstrated that the cage cavity had the potential to be a cost effective alternative to solid wall cavities. However, early RF spectrum measurements did not agree with the simulations. The cage cavity can approach RF properties of a solid wall cavity by using a coupled cavity design: The cage cavity is mounted in a large RF cavity in which this cavity's Eigen frequencies are decoupled from the cage cavity's Eigen frequencies. Computer models of the coupled cavity system will be presented to show that the quality factor of the cage cavity can be ~90% of the Q for a solid wall, superconducting cavity. The simulations also demonstrate several advantages of the cage cavity over a solid wall cavity, i.e. high order harmonic suppression, power coupling, and tuning. A prototype coupled cavity system has been fabricated and measurements of a cage cavity in a coupled cavity will be presented.

12:00pm **VT-WeM13 e-Cloud Activity of DLC Coated Chamber at FNAL Main Injector**, *Shigeki Kato*, KEK-High Energy Accelerator Research Organization, Japan, *J. Eldred*, Indiana University, *C.Y. Tan, M. Backfish, B. Zwaska*, FNAL

Carbon material that has a low mass density (resulting a long electron penetration depth in bulk), a low  $\delta_{\max}$  and a low secondary electron yield is of good option to mitigate electron cloud activity in particle beam chambers. In addition to this, diamond-like-carbon (DLC) coating on beam chambers would give advantages of a large deposition rate (a couple of  $\mu\text{m}/\text{h}$ ), inexpensiveness (~US\$800.- /m for coating of 100m), a good uniformity ( $\pm 5\%$ ) and coating applicability on any type of beam chamber (even bent one) and on any material without requirement of magnetic field. A DLC coated stainless steel chamber and a reference stainless chamber were installed into the FNAL main injector simultaneously in order to investigate e-cloud activity in the chambers with three retarding field analyzers (RFAs). Preliminary results at the low beam intensity showed RFA signals in the DLC coated chamber showed only 1/100 of that in the steel chamber. The e-cloud activity at the higher beam intensity and comparison of e-cloud activity at usually prepared surface (smooth surface) with roughed surface to aim further reduction of the activity will be also reported in the presentation.

# Wednesday Afternoon, November 12, 2014

## Vacuum Technology

Room: 303 - Session VT-WeA

### Accelerator and Large Vacuum Systems II

**Moderator:** James Fedchak, National Institute of Standards and Technology (NIST)

2:20pm **VT-WeA1 Load locks, Transfer arms, and other In-Vacuum Motions in the Cornell DC Photoelectron Gun Development Project.** *Karl Smolenski, X. Liu, B. Dunham, L. Cultrera, J. Conway*, Cornell University **INVITED**

The Cornell DC photoelectron guns pose a series of challenging vacuum engineering problems. These high brightness electron sources have produced the highest currents (0.075A) yet achieved from a photocathode source and are prototypes for future state of the art accelerators. The guns operate in the XHV (<1e-11 Torr) with massive NEG pumping and require exchange of the photocathode wafer periodically for continuous operation. The scale of the vacuum vessel, set by the extreme high voltages, requires the use of meter scale transfer arms to load and extract the photocathode holder with minimal disruption to the vacuum level and without particulate generation. These transfers are required to be rapid and simple to minimize operational downtime.

We have developed a series of mechanisms to retain the photocathodes, magnetic and bellows-coupled transfer arms to move samples between chambers, and load locks to introduce cathodes into the vacuum systems. More recently vacuum suitcases have been employed to move photocathodes from remote labs to our accelerators and to other laboratories for testing. This talk will present our experiences maintaining large scale systems with extensive in-vacuum motions under extreme requirements.

3:00pm **VT-WeA3 Vacuum Performance of 5-mm Undulator Chamber for Cornell High-Energy Synchrotron Source.** *Yulin Li, X. Liu, A. Lyndaker, A. Temnykh*, Cornell University

To significantly enhance the X-ray beam performance at Cornell High-Energy Synchrotron Source (CHESS), a 3.9-m long, 5-mm vertical aperture undulator vacuum chamber were designed, constructed and tested at Cornell Electron Storage Ring (CESR). The vacuum chamber is constructed of aluminum (Type 6061-T6) extrusions with an electron beam aperture (with nominal 5-mm vertical and 90-mm horizontal apertures), a pump antechamber and a cooling channel. To minimize the undulator magnet pole gap, pockets were machined on top and bottom of the extrusion in the middle portion. With the top and bottom wall thickness of 0.6 mm, the effective beam vertical aperture is reduced to 4.5mm owing to deflection from the atmospheric pressure. The undulator vacuum chamber was tested at its final designated location in CESR near a strong dipole magnet, intercepting high synchrotron radiation (SR) power and flux. To handle very high distributed gas load due to SR-induced desorption, six non-evaporable getter/ion pumps (NexTorr D100-5, SAES Getters) were installed along the undulator chamber. The test chamber was equipped with four cold cathode ionization gauges (CCGs) and a residual gas analyzer (RGA) to monitor vacuum performance. In this talk, we will present the construction, mechanical and vacuum qualifications, and the beam conditioning history of the undulator vacuum chamber. We will summarize the experiences learnt from the successful week-long beam tests.

3:20pm **VT-WeA4 Near-XHV Pressure Characterization for the Jefferson Lab Polarized Electron Source.** *Marcy Stutzman, P. Adderley, Thomas Jefferson National Accelerator Facility, M.A. Mamun, A.A. Elmustafa, Old Dominion University, M. Poelker, Thomas Jefferson National Accelerator Facility*

Long operational lifetime at the Jefferson Lab high polarization electron source requires vacuum approaching XHV ( $1 \times 10^{-10}$  Pa). Determining the ultimate pressure in a chamber requires minimizing outgassing rate, maximizing pumping, and accurately measuring pressure. Two systems were used to study the ultimate pressure that could be achieved: test chambers that were fabricated to characterize the effects on outgassing of different chamber material processing and coatings, and a cryopumped electron source sized chamber. This paper presents both the characterization of XHV gauges and the ultimate pressure achieved in the various chambers. The extent to which temperature dependent outgassing rate can be exploited to improve ultimate pressure will also be discussed. Finally, progress on reconciling the persistent discrepancies between calculated and measured pressure will be presented.

4:20pm **VT-WeA7 Design Optimization and Fabrication Progress of ITER's Large Custom Vacuum Pumps.** *Robert Pearce, M. Dremel, L. Worth, ITER Organisation, France, L. Baylor, S. Meitner, Oak Ridge National Laboratory* **INVITED**

ITER is under construction in the south of France in order to demonstrate the feasibility of fusion as a clean power source. It is one of the world's largest scientific and engineering collaborations. The civil structures, to house the ITER machine, are progressing, and the key systems and components are moving from design to manufacturing.

The ITER vacuum system will be one of the largest, most complex vacuum systems ever to be built. There are a number of large volume systems including: the cryostat (~8500m<sup>3</sup>), the torus (~1330 m<sup>3</sup>), the neutral beam injectors (~180m<sup>3</sup> each) and a number of lower volume systems including: the service vacuum system, diagnostic systems, and electron cyclotron transmission lines. In total there are more than 400 vacuum pumps of 10 different technologies required to pump the systems. The most demanding vacuum pumping applications are served by 18 large cryogenic pumps of 3 distinct custom designs.

The ITER vacuum vessel and cryostat are to be pumped by a total of 8 cylindrical cryo-sorption pumps with integral 800 mm all metal vacuum valves. The "build-to-print" design of these pumps has been optimised and finalised and the first pump is being manufactured.

The ITER neutral beam systems are each pumped by a pair of open structure panel style cryo-sorption pumps with a length of 8 m, and height of 2.8 m. They should achieve a pumping speed of 4500 m<sup>3</sup>/s for hydrogen. The final design of these pumps has involved development of new fabrication methods so as to significantly reduce the cost and manufacturing time for the thousands of cryo-panels and thermal shields within the pumps. The design is ready for manufacture, with the first pump destined for the ITER neutral beam test facility (MITICA).

During plasma operations, to pump the mixture of gasses originating from the regenerations of torus and neutral beam cryo-pumps, the roughing system will utilize 6 cryogenic viscous flow compressors (CVC). The principle of the CVC is that it will cryogenically condense hydrogen isotope mixtures, while providing first stage compression of helium ash originating from the fusion process. Each CVC is designed for throughputs of 200 Pam<sup>3</sup>/s and consists of a tube heat exchanger housed in a cryostat of diameter ~1 m and height 2.5 m. The very novel nature of this pump requires a full size prototype, which has been manufactured and will go through a test campaign.

In this paper an overview is given of the ITER construction. Examples of the cryo-pump 'value engineering' and design optimization for manufacturing are given. Progress and challenges in the "First of a Kind"(FOAK) vacuum pump manufacturing are given.

5:00pm **VT-WeA9 Commissioning of the KATRIN Main Spectrometer.** *Joachim Wolf, Karlsruhe Institute of Technology, Germany*

The objective of the Karlsruhe Tritium Neutrino experiment (KATRIN) at the Karlsruhe Institute of Technology (KIT) is the measurement of the electron neutrino mass with an unprecedented sensitivity of 200 meV by using electrons from the beta-decay of tritium. A central component is the electro-static main spectrometer (MS), where the energy of the beta-electrons (18.6 keV) will be measured with high precision. It consists of a large ultra-high vacuum vessel with a volume of 1240 m<sup>3</sup> and a surface of 690 m<sup>2</sup>, instrumented with a complex inner wire-based electrode system, which almost doubles the inner surface of the MS.

The pumping system of the MS consists of 6 turbo-molecular pumps (10 000 l/s), a large-scale getter pump (3000 m NEG strips, St707, 10<sup>6</sup> l/s) and three cryo-baffles (6.8 m<sup>2</sup>) at LN<sub>2</sub> temperature. The vacuum system has three major tasks: (I) the ultimate pressure, dominated by H<sub>2</sub>, has to be kept in the range of 10<sup>-11</sup> mbar in order to maintain a low background rate. (II) In conjunction with a differential pumping section and a cryogenic pumping section of the electron beam line, which connects the gaseous tritium source with the spectrometer, it has to keep the partial pressure of tritium in the MS below 10<sup>-21</sup> mbar. (III) The NEG strips are known to emanate a small amount of radon atoms, increasing the intrinsic background rate. Therefore cryogenic baffles at LN<sub>2</sub> temperature have been installed in front of the NEG pumps, which are expected to capture most of the radon atoms, before they can enter the sensitive volume of the MS. This paper describes the design of the vacuum system and reports on measurements of the vacuum performance during the first commissioning of the whole spectrometer system.

This work has been supported by the German BMBF (05A11VK3 and 05A11PM2).

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Pachicano, J.: VT-MoA8, 3; VT-MoA9, 3  
Park, C.D.: VT-TuA4, 8  
Pearce, R.: VT-WeA7, 13  
Poelker, M.: VT-WeA4, 13

## — R —

Rahman, O.: VT-TuP5, 10  
Rakowska, P.D.: AS+BI+VT-TuM6, 5  
Rao, T.: VT-TuP5, 10

Rebecchi, C.: VT-TuM12, 7  
Rees, J.A.: VT-TuP4, 10  
Ren, Y.: AS+BI+VT-TuM3, 5  
Ricker, J.E.: VT-MoM3, 1  
Ruck, R.L.: VT-TuA12, 9  
Ruiz, S.: VT-MoM5, 1  
Ruzic, D.N.: VT-MoA8, 3; VT-MoA9, 3

## — S —

Saeki, H.: VT-WeM6, 11  
Salter, T.L.: AS+BI+VT-TuM5, 5; AS+BI+VT-  
TuM6, 5  
Sanghera, J.S.: VT-MoA10, 3  
Scace, G.E.: VT-MoM3, 1  
Schlaf, R.: VT-TuM2, 6  
Seah, M.P.: AS+BI+VT-TuM6, 5  
Setina, J.: VT-MoM5, 1  
Seymour, D.L.: VT-TuP4, 10  
Sharipov, F.: VT-TuM5, 7  
Shchelkanov, I.A.: VT-MoA8, 3; VT-MoA9, 3  
Shrestha, B.: AS+BI+VT-TuM1, 5  
Siviero, F.: VT-TuM10, 7  
Skaritka, J.: VT-TuP5, 10  
Smith, T.L.: VT-WeM12, 12  
Smolenski, K.: VT-WeA1, 13  
Stillwell, B.: VT-WeM4, 11  
Stone, J.A.: VT-MoM3, 1  
Stopka, S.A.: AS+BI+VT-TuM1, 5  
Strouse, G.F.: VT-MoM3, 1  
Stutzman, M.L.: VT-WeA4, 13  
Swinney, T.C.: VT-MoM11, 2

## — T —

Tan, C.Y.: VT-WeM13, 12  
Temnykh, A.: VT-WeA3, 13  
Tridas, E.: VT-TuM2, 6

## — V —

Valente-Feliciano, A.-M.: VT-WeM10, 11  
Valizadeh, R.: VT-WeM1, 11  
Van de Runstraat, A.: VT-TuA10, 9  
Van der Donck: VT-TuM6, 7  
Van der Meer, J.A.: VT-TuA10, 9  
Van Groningen, T.: VT-TuA10, 9  
Versluis, R.: VT-TuA9, 9  
Vertes, A.: AS+BI+VT-TuM1, 5  
Viale, L.: VT-TuM10, 7  
Vicar, M.: VT-MoM5, 1  
Virgo, M.J.: VT-WeM12, 12

## — W —

Wang, E.: VT-TuP5, 10  
Wang, L.: VT-TuA11, 9  
Wang, X.: AS+BI+VT-TuM3, 5  
Wells, M.: AS+BI+VT-TuM12, 6  
Wilson, K.: VT-WeM3, 11  
Wiseman, J.W.: AS+BI+VT-TuM10, 6  
Wolf, J.: VT-WeA9, 13  
Worth, L.: VT-WeA7, 13  
Wüest, M.P.: VT-MoA1, 3

## — X —

Xia, Y.: AS+BI+VT-TuM3, 5  
Xie, X.: VT-TuA12, 9  
Xu, H.: VT-WeM3, 11

## — Y —

Yoshida, H.: VT-MoM8, 2  
Yu, C.S.: VT-TuP2, 10  
Yu, M.: VT-MoM1, 1

## — Z —

Zhang, L.: AS+BI+VT-TuM1, 5  
Zigrosser, D.: VT-WeM3, 11  
Zou, R.: AS+BI+VT-TuM3, 5  
Zwaska, B.: VT-WeM13, 12

